

Development and Evaluation of a Carbon Filter for Removing DMSO Vapor from the Exhaust of the W79 HE Dissolution Workstation

W. Bergman
K. Wilson
G.O. Nelson

August 1998



This is an informal report intended primarily for internal or limited external distribution. The opinions and conclusions stated are those of the author and may or may not be those of the Laboratory.

Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

DISCLAIMER

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

This report has been reproduced
directly from the best available copy.

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information
P.O. Box 62, Oak Ridge, TN 37831
Prices available from (423) 576-8401

Available to the public from the
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Rd.,
Springfield, VA 22161

DEVELOPMENT AND EVALUATION OF A CARBON FILTER FOR REMOVING DMSO VAPOR FROM THE EXHAUST OF THE W79 HE DISSOLUTION WORKSTATION *

by

W. Bergman, K. Wilson, and G. O. Nelson¹
Lawrence Livermore National Laboratory
Livermore, CA 94550

Abstract

We have developed and evaluated a carbon filter to remove dimethyl sulfoxide (DMSO) vapor from the exhaust of the W79 HE (high explosive) Dissolution Workstation. A series of laboratory tests were conducted on eight different carbon samples to establish the vapor breakthrough curves and to select the best sample. Empirical equations were developed to predict the breakthrough curve for various air flows and bed thickness. These equations were used to design the filter cartridge. The filter consists of a cylindrical cartridge 12 inches high and 6 and 10 inches inside and outside diameter and has a pressure drop of 0.8 inches of water at 50 cfm. Experiments and computations verified the efficiency of the filter cartridge is greater than 99% at 35 cfm, and the carbon filter can process 60 HE assemblies while maintaining greater than 90% efficiency before the carbon must be replaced.

Introduction

This study describes the development of a carbon filter that is a component of the exhaust system for the W79 HE Dissolution Workstation as shown in Figure 1. The Workstation is designed to support the DOE nuclear weapons dismantlement program in which hot DMSO sprays are used to dissolve the HE from HE assemblies. The description of the W79 Workstation and the complete air cleaning system is given in a previous report ⁽¹⁾. We developed the carbon filter because commercially available units were either designed for much larger ventilation systems or had excessive pressure drops. The typical ventilation filter has a 2' x 2' cross sectional area and is designed for flow rates of 1,000 cfm or greater. Smaller annular cartridge filters were developed for military vehicle applications but have pressure drops that greatly exceed the requirement of less than one inch of water at 50 cfm. In addition to the lack of suitable carbon filter units, there also is no information on the performance of carbon filters for removing DMSO vapors. Thus, we had to establish the basic adsorption characteristics in addition to the design parameters to develop the DMSO carbon filter. The initial step was to demonstrate that activated carbon was effective in removing DMSO vapors.

*This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48.

¹ Miller-Nelson Research, Inc. 8 Harris Court, Suite C6, Monterey, CA, 93940

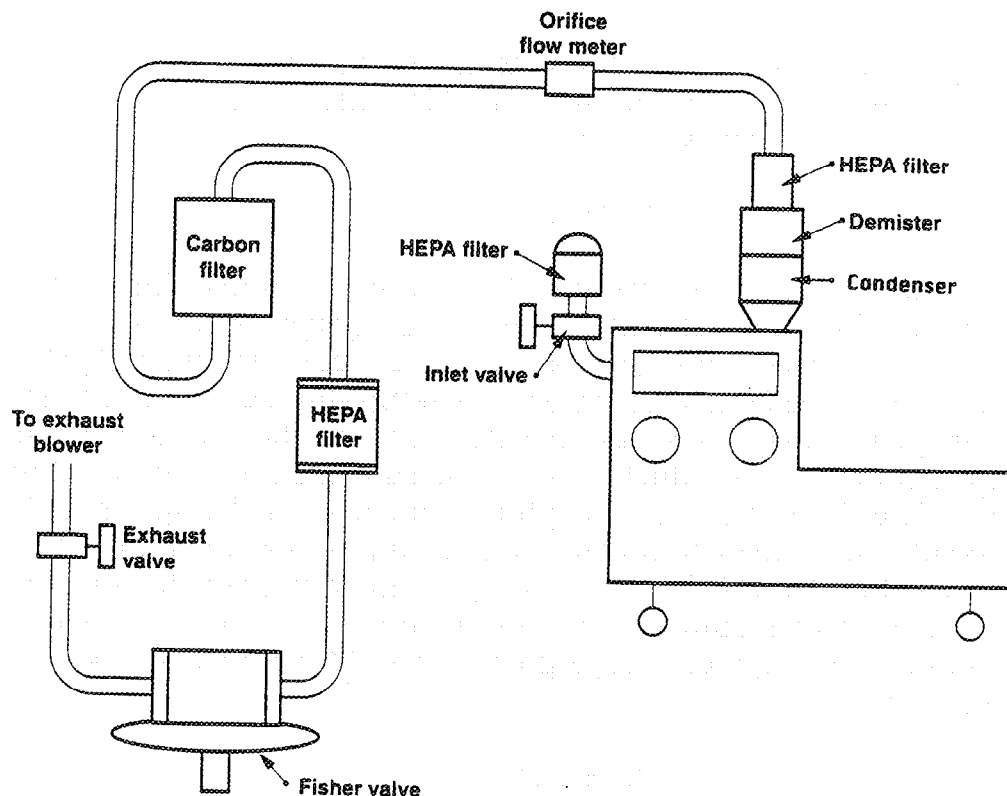


Figure 1. Schematic of exhaust system on the W79 HE Dissolution Workstation.

Small Scale Laboratory Studies

We conducted a survey test of eight different activated carbons to establish that activated carbon is effective in removing DMSO vapors and to identify the best carbon. The test carbons were packed into empty respirator cartridges having a total carbon volume of 112 cc, a diameter of 7.9 cm and a bed depth of 2.3 cm. The cartridges were filled with carbon poured through a tube having multiple screens and a 2 feet free fall to insure tight packing. Each of the cartridges was weighed before and after the test to establish the weight of the carbon and the weight gain due to adsorbed DMSO vapor. The test consisted of passing 29.4 l/min air containing 500 ppm (1.59×10^{-3} g/l) of DMSO vapor with less than 10% relative humidity at 25 °C through each cartridge. The DMSO vapor was generated by injecting a known flow of liquid DMSO using a calibrated syringe pump into a heated block that vaporized the liquid into a controlled air flow. The airflow rate corresponded to a flow velocity of 6 m/min since the area was 49

cm² (4.9 l/m). The airflow, gas temperature, and relative humidity were controlled with a Miller-Nelson flow control module. The efficiency of DMSO removal was determined by measuring the concentration of DMSO vapor before and after the cartridge using a Miran infrared analyzer (Foxborough Inc.). Since each of the tests was conducted until the carbon was saturated and had 0% efficiency, the increase in carbon weight represented the dynamic adsorption capacity of the carbon for DMSO. Figure 2 shows a schematic of the experimental apparatus used to conduct the small-scale carbon tests.

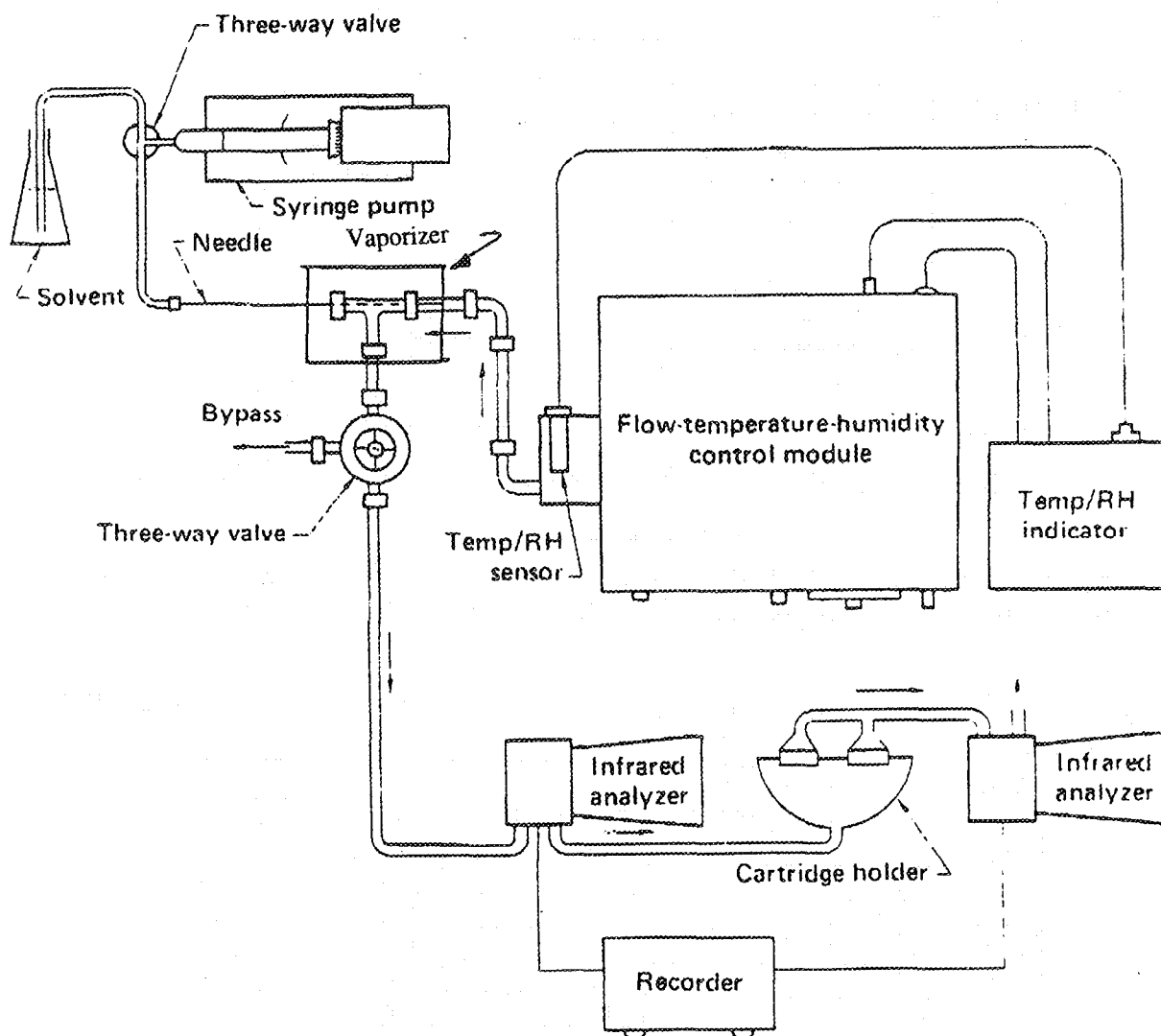


Figure 2. Schematic diagram of the experimental apparatus used in the small-scale carbon tests.

Figure 3 shows the DMSO removal efficiency for each of the eight carbon canisters as a function of exposure time. All of the carbons have similar DMSO adsorption properties. However, sample G215 from PICA USA Inc. (Columbus, OH) had a slightly better performance than the other samples and was therefore selected for the carbon filter in our project. The carbon weight, adsorbed DMSO weight, and the pressure drop for each of the canisters is shown in Table 1.

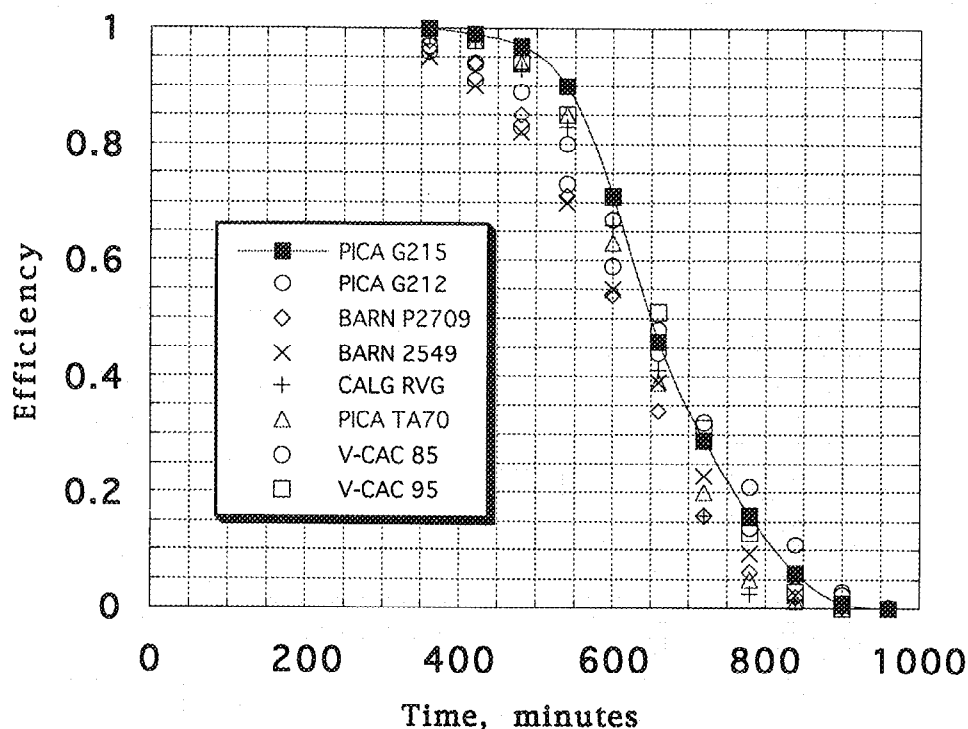


Figure 3. Survey of carbon capacities plotted as efficiency versus exposure time at 6m/min flow velocity and 500 ppm (1.59 g/m³) DMSO.

Table 1. Experimental measurements of carbon cartridge samples in screening tests at 6 m/min.

Sample	Carbon Weight, g	DMSO Adsorbed, g	Pressure Drop, in. H ₂ O
PICA G215	45.9	31.6	0.31
PICA G212	42.6	31.3	0.21
BARN P2709	55.2	29.7	0.27
BARN 2549	47.7	30.4	0.20
CALG RVG	51.7	30.9	9.31
PICA TA70	51.1	30.6	0.30
V-CAC 85	44.3	31.9	0.29
V-CAC 95	45.8	32.5	0.31

We then conducted additional tests on new cartridges of PICA G215 at 14.7 and 43.9 l/min flow rates, which correspond to flow velocities of 3 and 12 m/min. These tests were needed to establish the adsorption parameters for the design of

the carbon filter. Figure 4 shows the results of the three carbon canisters of PICA G215 tested at 3, 6 and 12 m/min flow velocity. We see that the faster flows results in shorter saturation times. The experimental measurements of the PICA G215 carbon samples are shown in Table 2.

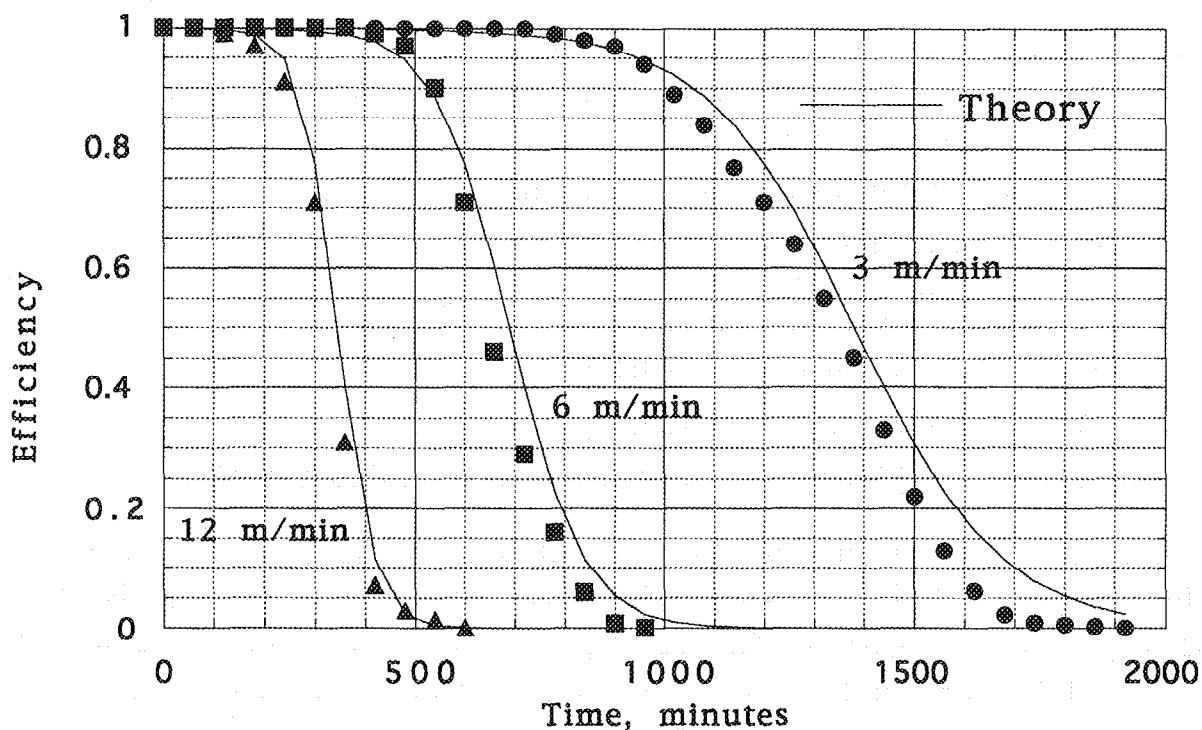


Figure 4. Efficiency of PICA G215 canisters at 3, 6 and 12 m/min flow velocity. Points are experimental data; solid lines represent theory.

Table 2. Experimental measurements of PICA G215 carbon samples in tests at 3, 6 and 12 m/min.

Velocity V m/min	Flow rate, l/min	Pressure drop in. H ₂ O	Carbon Wt W _c , g	DMSO Wt W _a , g	Saturated Capacity W _s =W _a /W _c	Carbon bulk density, ρ_c g/l
3	14.7	0.13	44.0	32.8	0.745	393
6	29.4	0.31	45.9	31.6	0.688	410
12	58.8	0.57	43.9	32.5	0.740	392

In order to design an activated carbon filter for removing DMSO vapor, it was first necessary to determine the adsorption capacity of the selected carbon for DMSO, W_s , and the coefficient, K_v , for the rate of adsorption. Once these two parameters are determined, theoretical equations can be used to determine the key design parameters for the gas filter. The dynamic adsorption capacity, W_s , was experimentally determined and is shown in Table 2. The kinetic coefficient, K_v , characterizing the rate of adsorption is derived from matching experimental

data with a theoretical model. We used the modified Wheeler equation, shown in Equation 1 to derive the kinetic coefficient.⁽²⁾

$$t = W_s W_c / (C A V) + W_s \rho_c / (C K_v) \ln [(1 - E) / E] \quad (1)$$

where

- A = cartridge area, 4.9 l/m (49 cm²)
- C = DMSO concentration, 1.59 x 10⁻³ g/l (500 ppm)
- E = DMSO removal efficiency
- K_v = kinetic adsorption coefficient, min.⁻¹
- ρ_c = carbon bulk density, g/l
- t = time, minutes
- V = flow velocity, m/min.
- W_c = carbon weight in cartridge, g
- W_a = DMSO weight adsorbed, g
- W_s = DMSO saturated capacity = W_a/W_c, dimensionless

Values of K_v were determined by substituting the parameter values from Table 2 into Equation 1 and fitting each of the three equations to the corresponding efficiency curves in Figure 4. The least squares fits produced K_v values of 1369, 2750 and 4801 min⁻¹ at 3, 6 and 12 m/min flow velocities respectively. To establish the velocity dependence of K_v, we plotted the values of K_v versus the flow velocity in Figure 5.

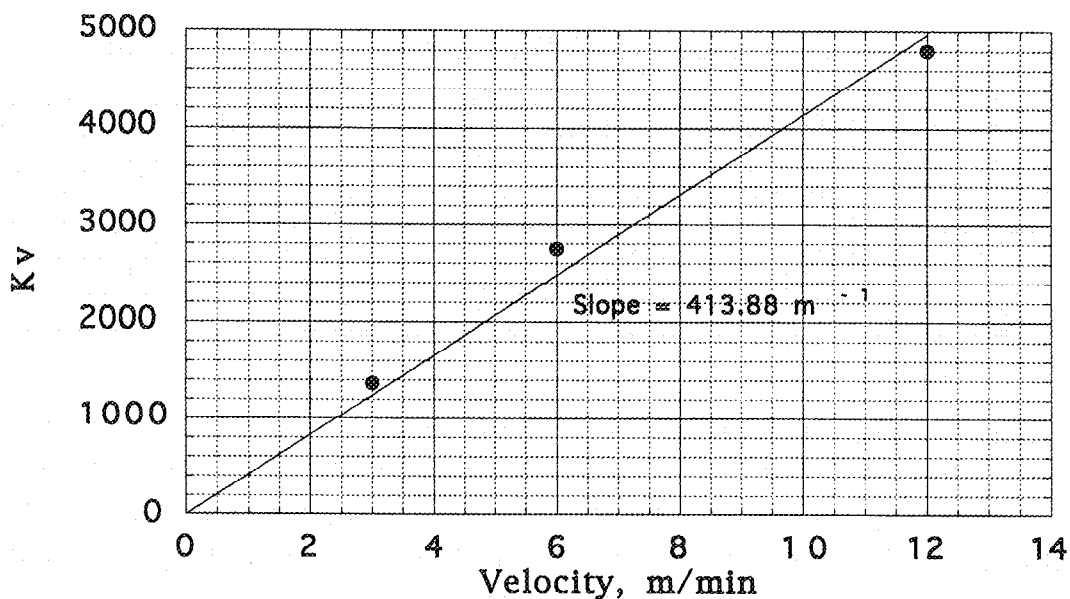


Figure 5 Determination of K_v dependence on velocity.

From Figure 5, we have

$$K_v = k_v V = 413.88 \text{ V min.}^{-1} \quad (R=0.99035) \quad (2)$$

where the slope, k_v , equals 413.88 m^{-1} . Substituting this expression into Equation 1 yields,

$$t = W_s W_c / (C A V) + W_s \rho_c / (C k_v V) \ln [(1 - E) / E] \quad (3)$$

All of the constants in Equation 3 can be lumped together into constants c_1 and c_2 to yield.

$$t = c_1 / V + (c_2 / V) \ln [(1 - E) / E] \quad (4)$$

Replacing the constants in Equation 4 with the values in Table 2 yields $c_1 = 4207.4, 4053$ and 4169.7 and $c_2 = 444.6, 427.7$ and 439.7 for $V = 3, 6$ and 12 m/min respectively. Averaging these constant values yields

$$t = 4143.4 / V + (437.3 / V) \ln [(1 - E) / E] \quad (5)$$

Equation 5 was used to generate the theoretical curves in Figure 4.

The increasing deviation in Figure 4 between the experimental data and theory at slower velocities is most likely due to the assumption of irreversible adsorption. In practice, the adsorbed DMSO will not be adsorbed irreversibly and will be released, which is more pronounced at longer times and at higher carbon saturation values.

Design of Full-scale Carbon Filter

We used Equation 5 and the parameter values determined in the cartridge studies to design the carbon filter for use in the W79 HE Dissolution Workstation. A major constraint on the filter design is the thickness of the carbon bed. Although we used a bed depth of 2.3 cm (0.91 inch) in our screening tests, existing standards on carbon filters require a minimum of 2 inches^(3,4). This minimum bed depth was established to avoid channeling effects in the ventilation filters. However, since the pressure drop across the filter is directly proportional to the bed depth, it is desirable to minimize the bed depth. Thus, the optimum bed depth that satisfies the current standards and minimizes the pressure drop is 2 inches. To include the effect of carbon bed depth in Equation 3, we made the following two substitutions:

$$Q = A V \quad (6)$$

$$\rho_c = W_c / A T \quad (7)$$

where, Q = flow rate, l/min.
 T = carbon bed thickness, m

Substituting Equations 6 and 7 into Equation 3 and rearranging yields,

$$t = W_s W_c / (C Q) [1 + (1 / k_v T) \ln [(1 - E) / E]] \quad (8)$$

Equation 8 can be used to establish how much carbon, W_c , should be used for a given exhaust flow rate, Q , and concentration of DMSO, C and for a desired filter life, t . However since the DMSO concentration and exhaust flow rate vary considerably during a typical dissolution operation, it is more efficient to size the carbon filter based on the estimated total mass of DMSO vapor from the dissolution and clean-up. The total mass, M , of DMSO vapor exhausted from the Workstation for a single HE assembly is computed from the following equation:

$$M = \sum n_i C_i Q_i t_i \quad (9)$$

where i = the i th phase of the dissolution operation.

n_i = the number of cycles for a given operation.

Substituting Equation 9 into Equation 8 yields

$$N = (W_s W_c / M) [1 + (1 / k_v T) \ln [(1 - E) / E]] \quad (10)$$

where N = number of HE assemblies that can be cleaned before replacing the carbon filter.

Substituting the values $E = 0.9$, $k_v = 413.9 \text{ m}^{-1}$, $T = 0.0508 \text{ m}$, we have

$$N = (W_s W_c / M) (0.896) \quad (11)$$

We can estimate the mass of DMSO vapor, M , entering the carbon filter from Equation 9 for the different stages of the dissolution operation. We estimated the DMSO vapor concentration, C_i , from the equilibrium vapor concentration versus temperature in Figure 6⁽⁵⁾. The corresponding temperature is measured after the demister pad in the Workstation exhaust system. The temperatures during the

dissolution operation were experimentally determined in early tests at LLNL. The temperatures for the rinse operations are estimates. The exhaust flow is experimentally determined to be about 1 cfm (due to leaks) during the non-vented operations and 35 cfm in the vent mode when the inlet valve is open. The measured times also represent early tests at Pantex. However, since LLNL tests showed the Workstation aerosol concentration was reduced to nearly 0 after 2 minutes with a 20 cfm flow, we estimate the Workstation will be purged within 1 minute at 35 cfm. In addition, we used one half of the peak vapor concentration to represent the average during this period. All of the values for C_i , Q_i , t_i , and n_i are shown in Table 3. From Table 3 we have $M = 42.3$ g.

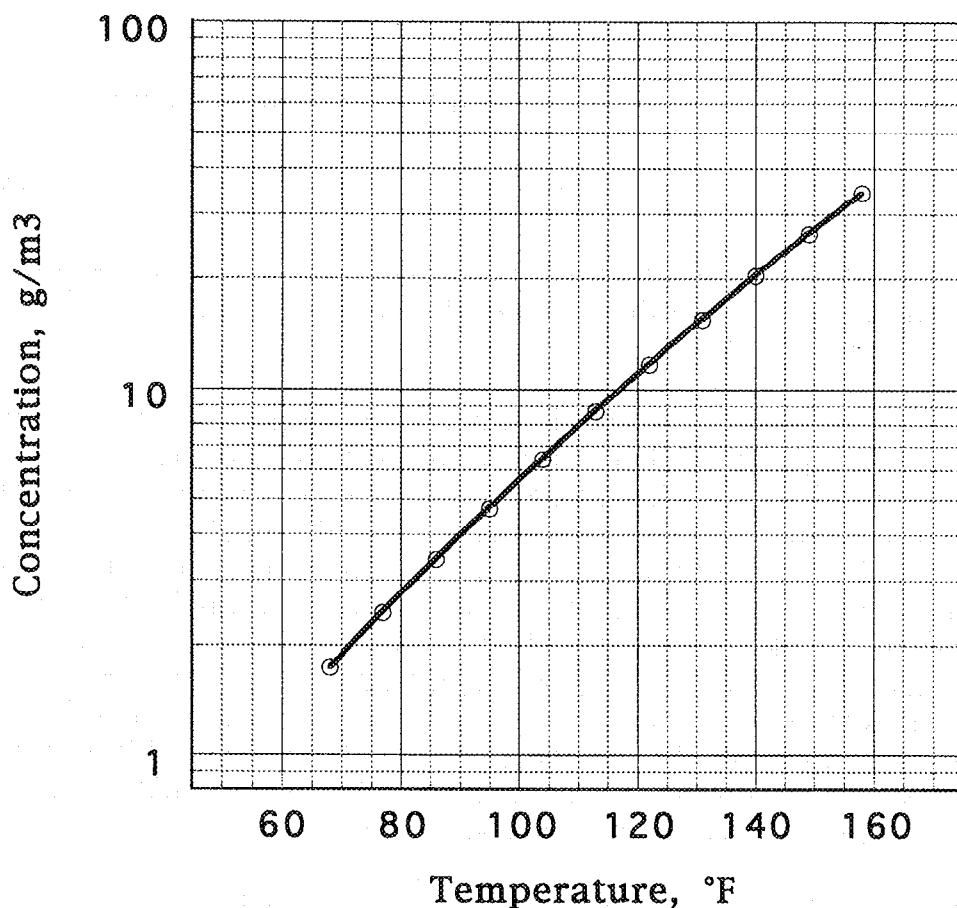


Figure 6. Mass concentration of DMSO vapor corresponding as a function of temperature ⁽⁵⁾.

Table 3. Determination of DMSO vapor from Equation 9.

Operation	Temp °F	DMSO Conc. (C_i) g/m ³	Exhaust flow, (Q_i) cfm	Time (t_i) Min.	Number of cycles n_i	DMSO mass, (m_i) g
Dissolution/spray	65	1.5	1	360	1	15.3
Dissolution/vent	60	0.6	35	60	24	14.3
Rinse/ SprayManifold	60	1.2	1	120	1	4.1
Rinse/Workstation	65	1.5	1	30	1	1.3
Rinse/ Vent	60	0.6	35	1	5	3.0
Condensor/rinse	90	4.0	1	30	1	3.4
Condenser/vent	70	0.9	35	1	1	0.9

Substituting the values $M = 42.3$ g and $W_s = 0.724$ into Equation 11 yields,

$$N = 0.0153 W_c \quad (12)$$

Equation 12 shows the number of HE assemblies increases in direct proportion to the mass of carbon in the carbon filter. Each HE assembly requires 65.3 g of carbon.

Packaging the carbon into a filter element was constrained to having a 2 inch thick bed to minimize the pressure drop and minimizing the overall size while providing at least one month of continuous operation. We selected the cylindrical cartridge design because of the ease of fabrication and refilling the carbon. The final dimensions selected were 6 and 10 inches inside and outside diameters and 12 inches high. This unit has a volume of 9.88 l and can accommodate 3930 g of carbon. Based on Equation 14, this carbon filter can be used to process about 60 HE assemblies (Equation 12 shows 59.7) before replacing the carbon. We should note that more accurate estimates of carbon life could be obtained with improved input data in Table 3. Alternatively, the efficiency of the carbon filter can be measured experimentally at increasing loadings to establish the actual number of HE assemblies that can be processed prior to carbon replacement.

The schematic of the cylindrical cartridge filter that we designed is shown in Figure 7, where the cartridge is shown mounted inside a cylindrical housing. The exhaust flows from the bottom port into the interior of the carbon filter, passes through the 2 inch bed and exits around the exterior of the cartridge and finally through the top port. The carbon bed is a 2-inch thick concentric cylinder contained between concentric cylinders made of perforated metal screens that are 12 inches long and have 10 and 6-inch diameters. The net carbon volume of this

filter is 9.88 l. Figures 8-10 show the partially assembled canister, the fully assembled canister, and the exhaust system housing for the canister.

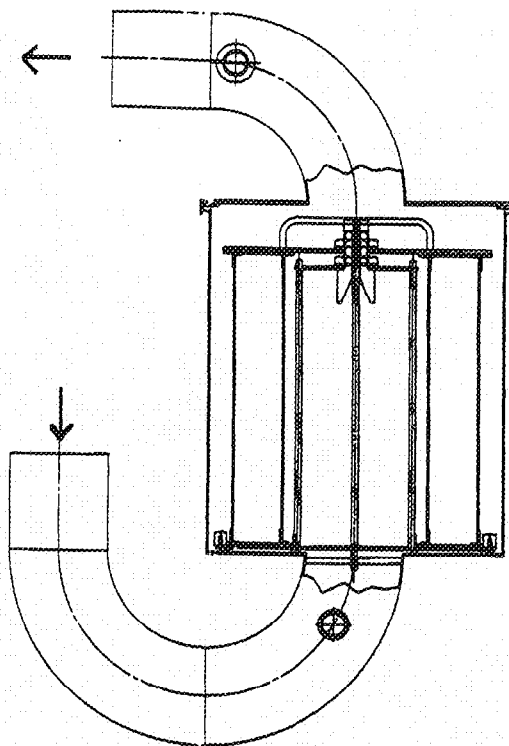


Figure 7. Schematic of cylindrical carbon filter cartridge mounted in a housing.

To prevent the possibility of DMSO condensing and collecting inside the carbon filter, we directed the exhaust through a U-tube section before entering the carbon filter. Any DMSO condensate would collect at the low point of the U-tube and can be drained through a plug. It is possible to form DMSO condensate in the lines when the ambient laboratory temperature is lower than the exit temperature of the demister. This may occur when rinsing out the condenser because hot DMSO liquid is sprayed on the condenser tubes from above. Hot DMSO vapor will therefore pass into the exhaust system without cooling in the heat exchanger. As the heated DMSO vapor cools, it will condense in the exhaust lines and filters.

We measured the efficiency of the carbon filter during a practice dissolution operation and showed the carbon filter was only about 70% efficient. We measured the DMSO concentration before and after the filter with a Boxborough infrared analyzer. Since the predicted efficiency should be in excess of 99%, we suspected the poor performance was due to the carbon loading procedure that we used. We filled the annular space with carbon by pouring the carbon granules directly from a container and then shaking the filter unit to insure tight packing. This test demonstrated the importance of using the established carbon filling procedure where carbon granules are poured through multiple screens to insure a well packed bed.

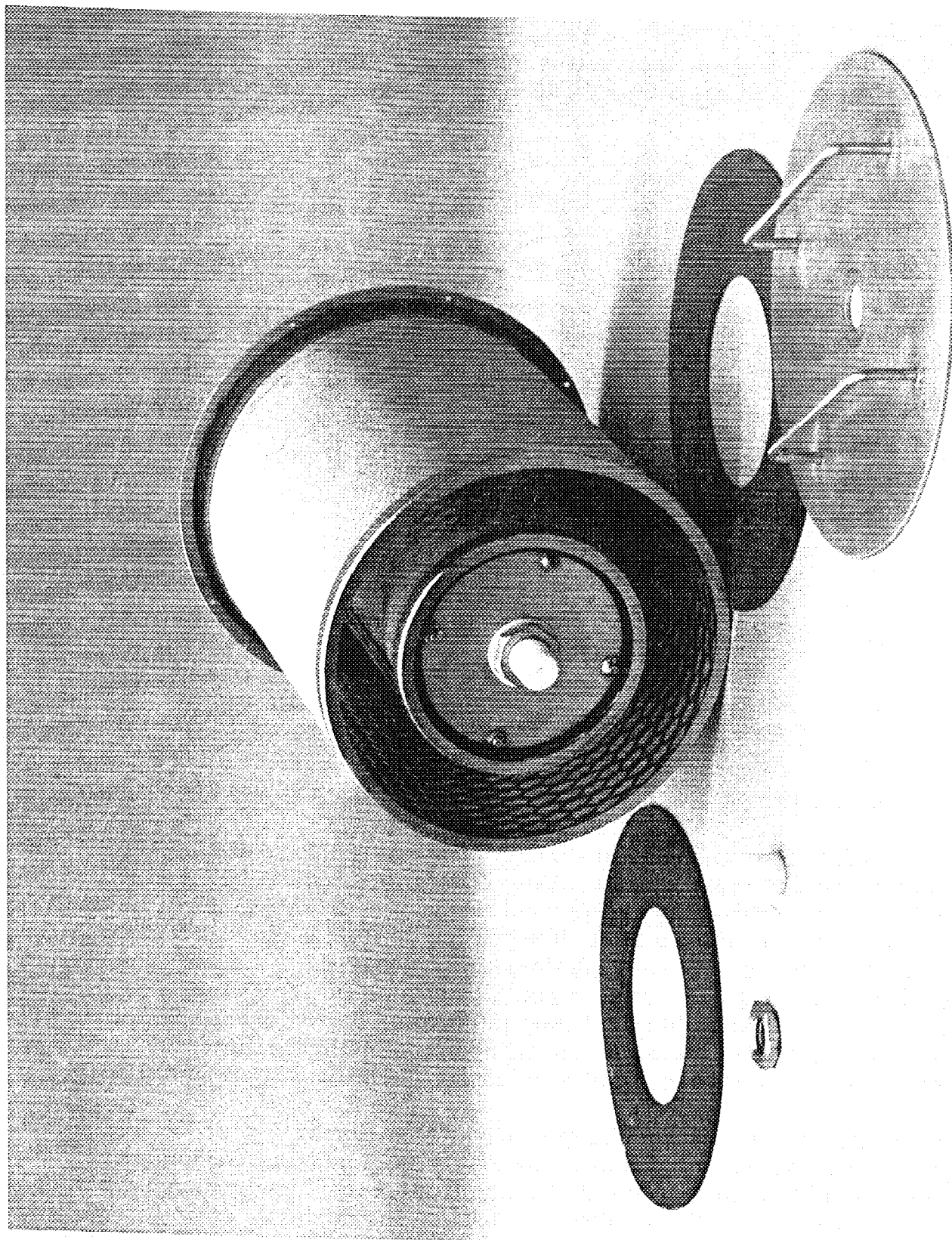


Figure 8. Photograph of the cylindrical filter canister partially disassembled showing the annular space for holding the granular carbon. The view is from the topside.

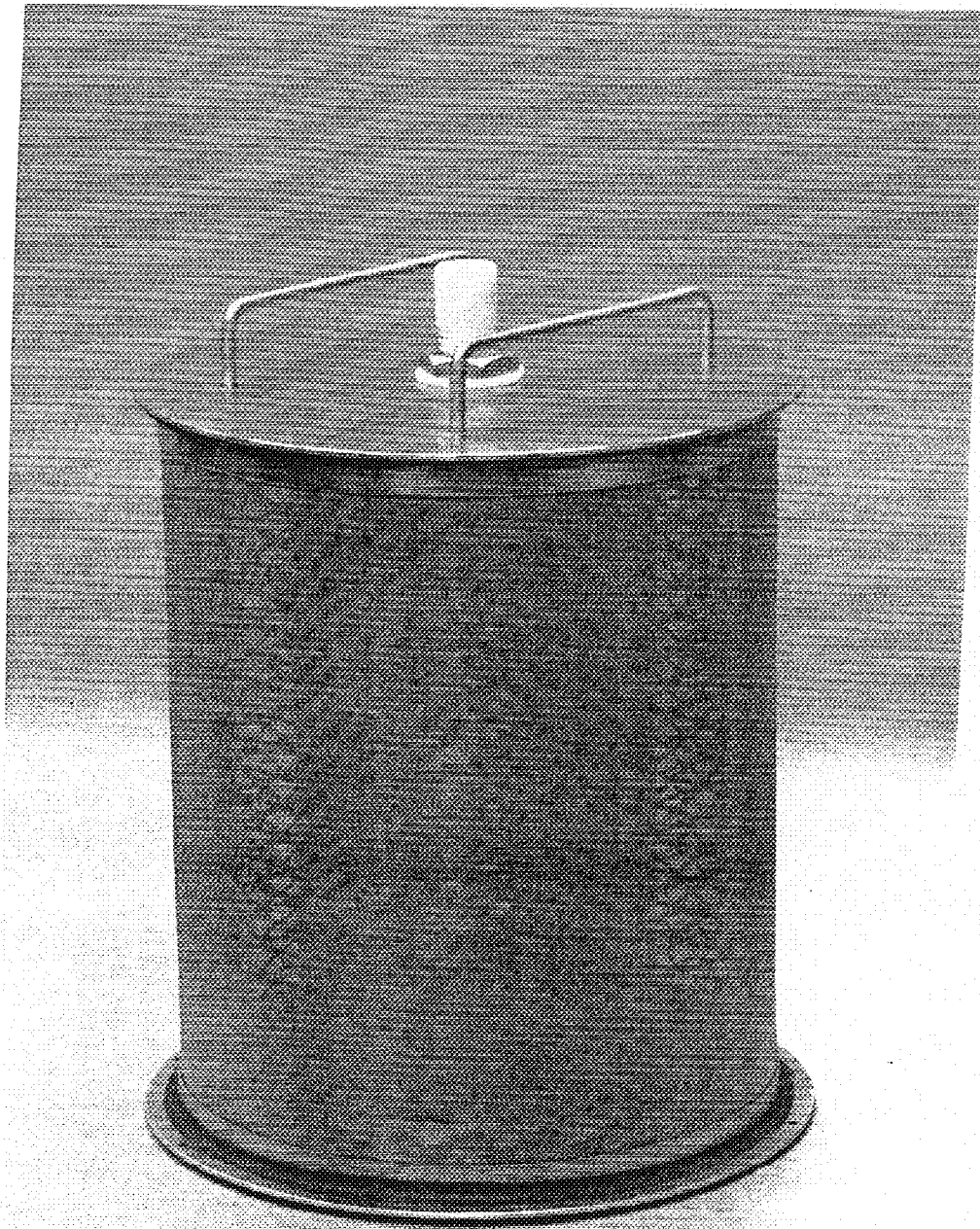


Figure 9. Photograph of the assembled carbon filter canister.

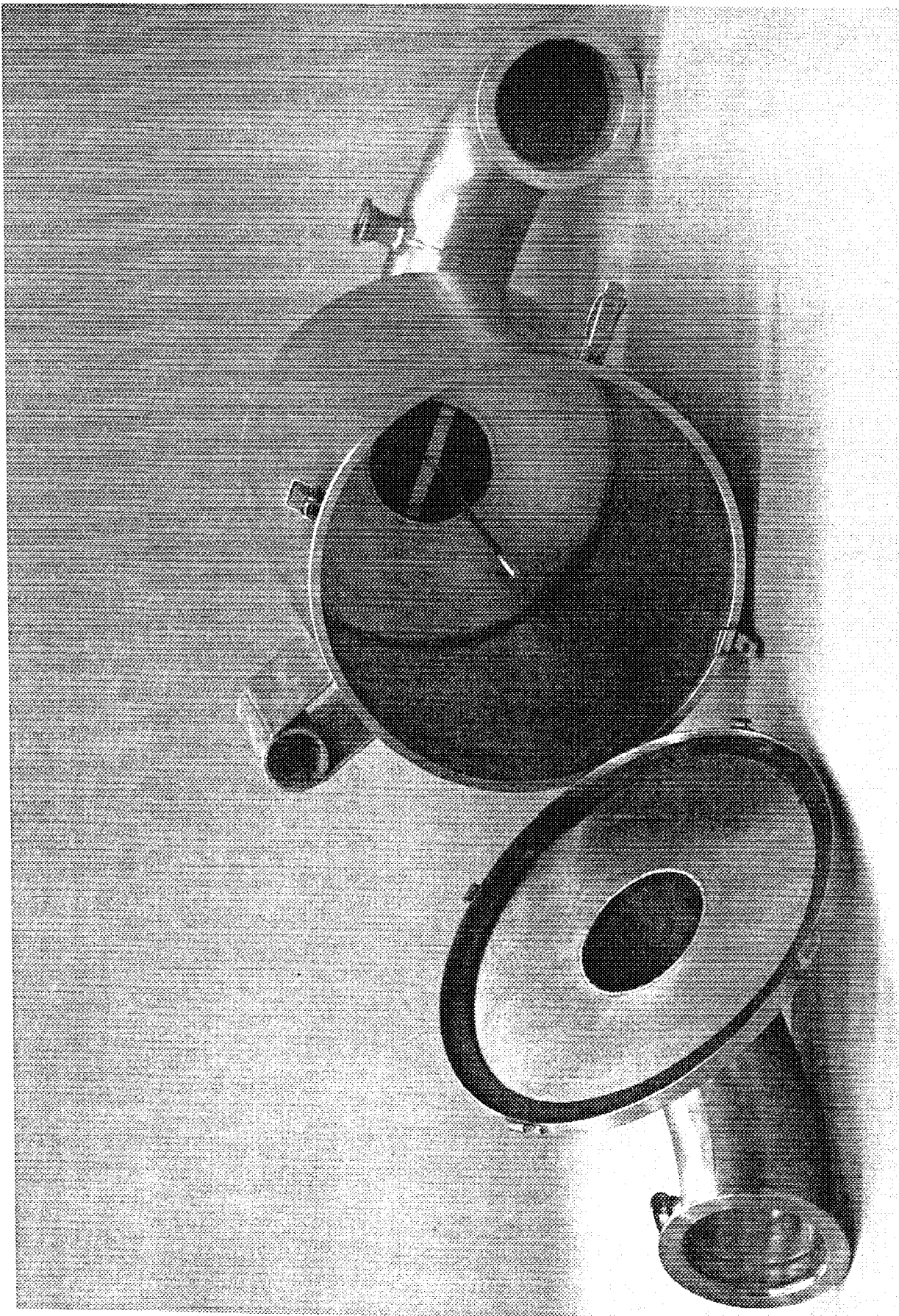


Figure 10. Photograph of the exhaust system housing for the carbon canister.

Filling Canister With Carbon Granules

We designed and built a filling tube, shown in Figure 11, to be used when filling the carbon canister with carbon granules. The filling tube has an inlet nozzle and two stages of four screens with 1/4" x 1/4" openings. The screens act to disperse the carbon granules in a uniform distribution across the area independent of the initial point of entry where the carbon is poured. The first set of screens functions to disperse the carbon granules across the 2-inch inlet tube so the carbon is dispersed uniformly in a radial position by the cone. The second set of screens functions to disperse the carbon granules uniformly within the annular space of the carbon canister. Note the carbon filling tube fits directly over the carbon canister in Figure 11.

The procedure for filling a fresh charge of carbon into the canister is illustrated in Figures 12-14. The first step is to open the top lid of the canister and remove the old carbon. The empty canister and filling tube are shown in Figure 12. The filling tube is then mounted over the canister as shown in Figure 13. Carbon granules are then poured into the inlet funnel as illustrated in Figure 14. Additional carbon is added until the annular space of the canister is filled slightly over the rim. After the canister is filled, the inlet tube is lifted off the canister, and the carbon deposits inspected to verify the deposits are uniform around the circumference and are slightly over the top surface. A rubber gasket is then placed over the annular space and the top plate fastened to the canister

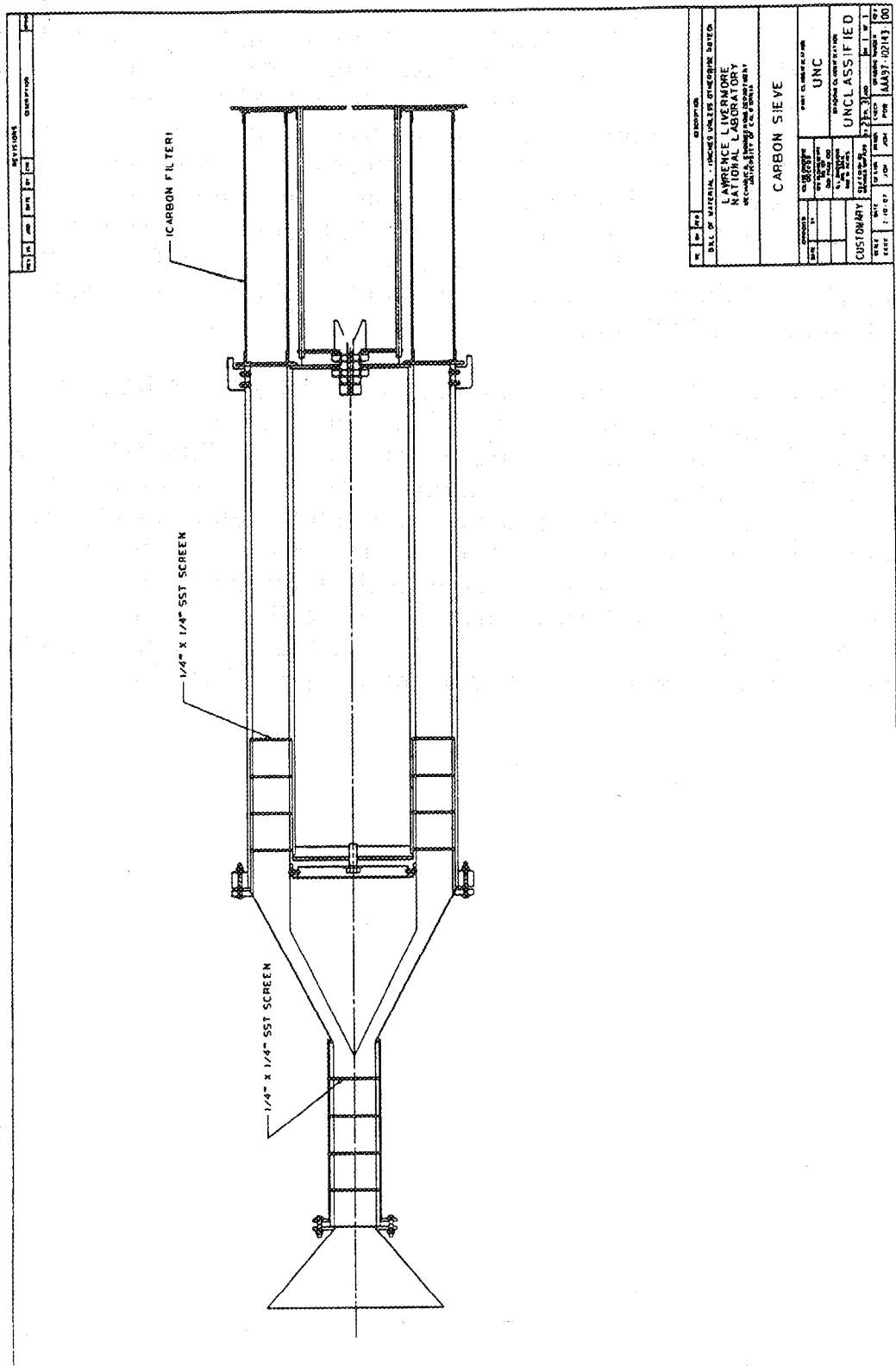


Figure 11. Schematic of carbon filling tube mounted over the carbon canister.

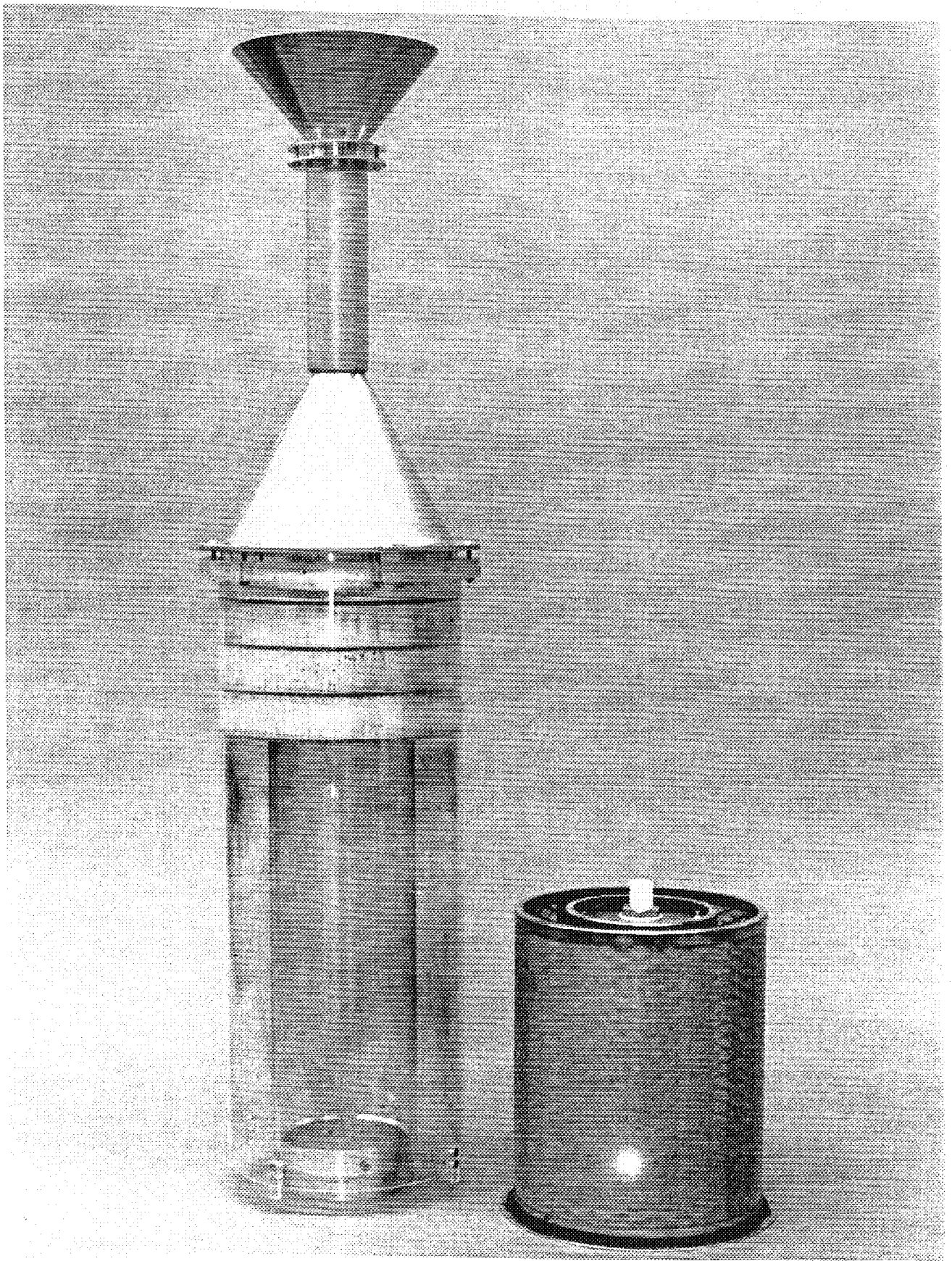


Figure 12. Photograph of the carbon filling tube and the empty carbon canister.

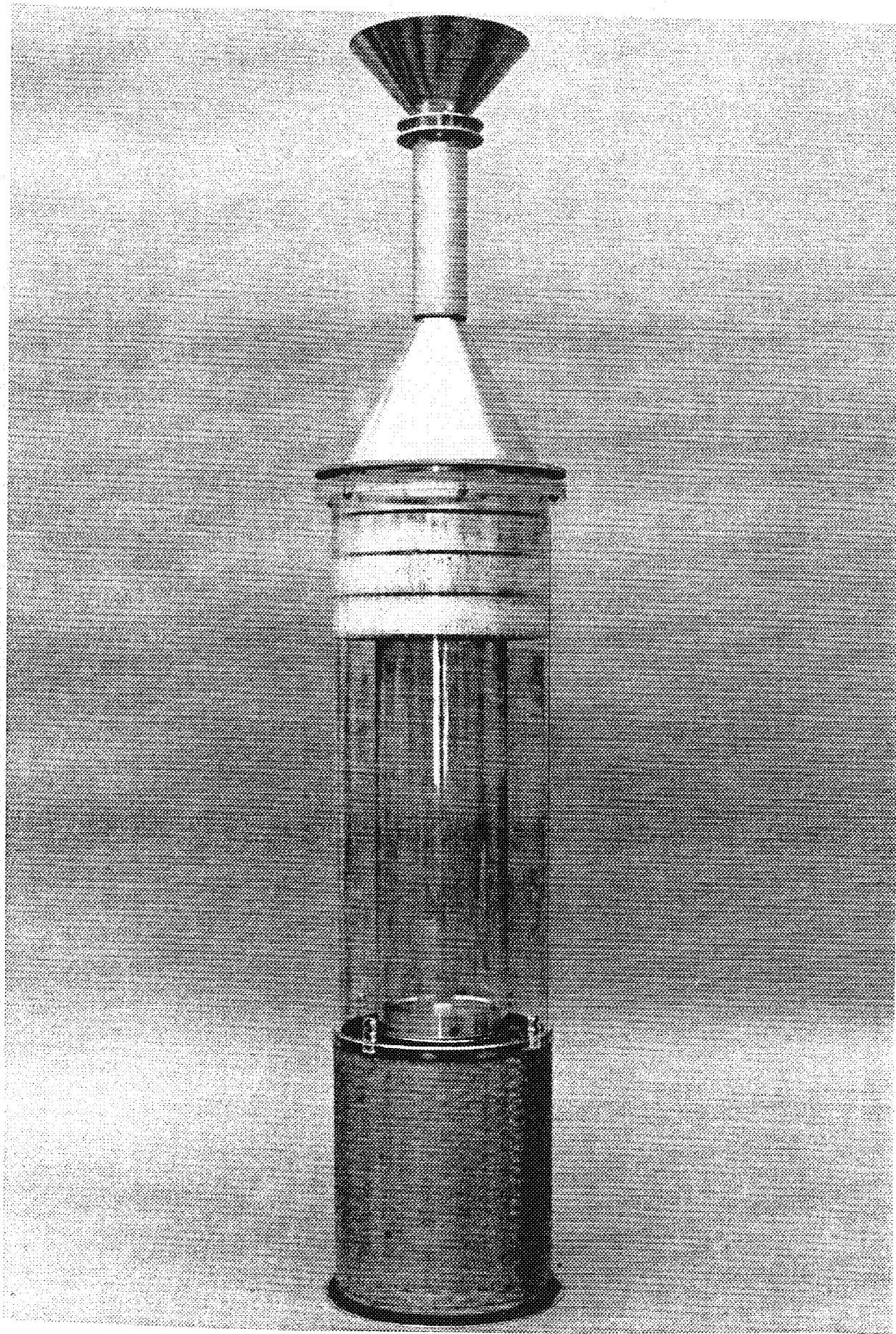


Figure 13. Photograph of the carbon filling tube mounted on the carbon canister.

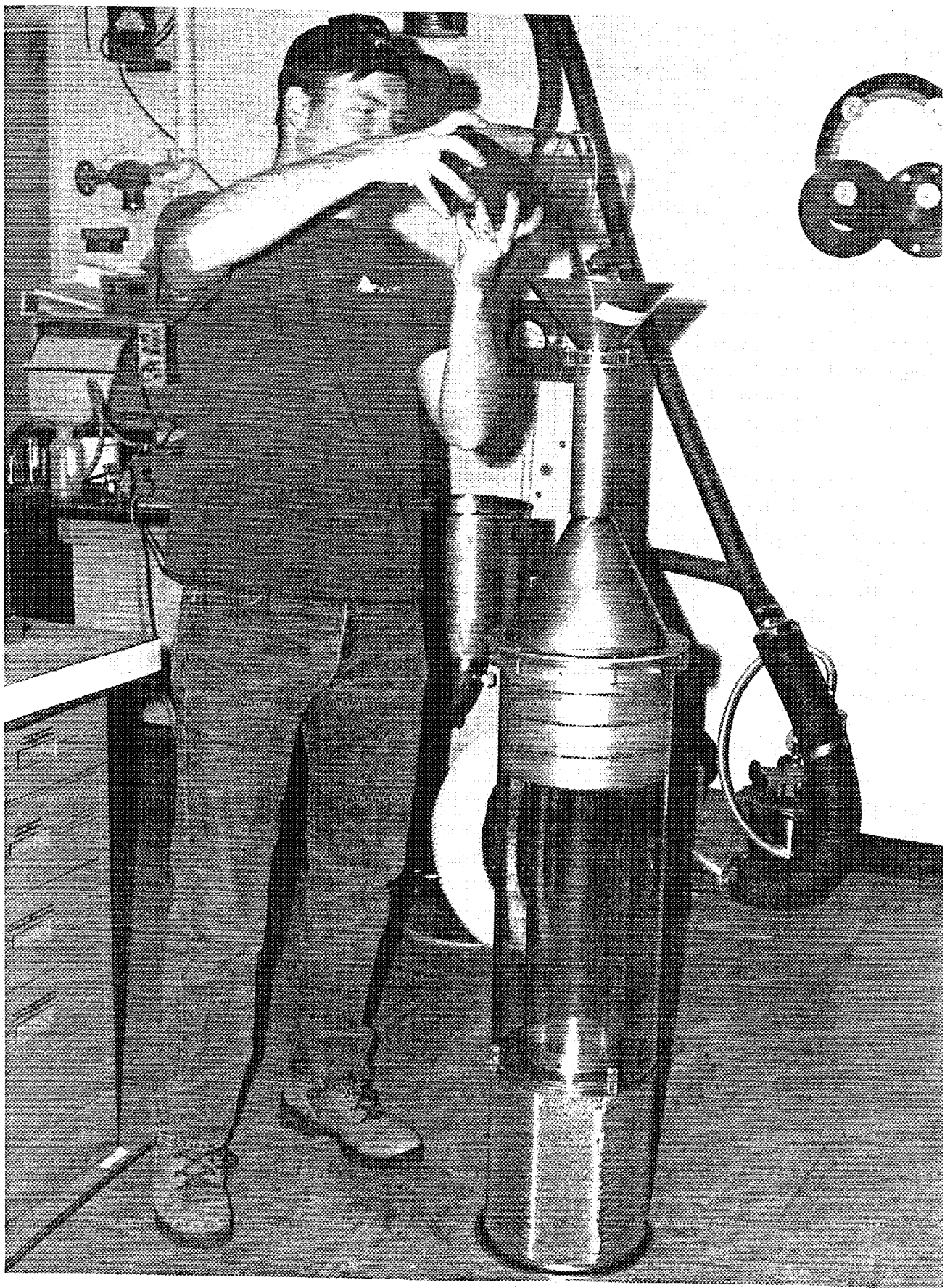


Figure 14. Photograph illustrating the use of the filling tube to fill the carbon canister with carbon .

Measurement of Carbon Canister Efficiency

We measured the efficiency of the carbon canister mounted inside the exhaust system housing in a similar fashion as was done with the screening tests on small-scale tests. However, we measured only the efficiency of a fresh canister because of time limitations to conduct the full breakthrough curve as shown in Figure 4 for the small-scale filter. Using Equation 8, we estimate it would take about 40 hours of exposure to measure the breakthrough curve at the maximum room temperature exposure (500 ppm, 1.6 g/m³) and a flow rate of 35 cfm. Moreover, measuring the breakthrough curve for the carbon canister is not sufficient to establish the filter life in the W79 dissolution operation because of the variability in concentration, flow rate, and exposure time at each condition. The primary purpose of the efficiency test on the carbon canister is to demonstrate the efficacy of the carbon filling technique.

The schematic of the experimental apparatus used for measuring the efficiency of the carbon canister is shown in Figure 15. The apparatus is similar to that used for the small scale evaluations except no conditioned air supply is used to generate the 35 cfm. A syringe pump injects liquid DMSO into a 7 cfm, secondary air flow that is then directed into the 35 cfm air flow passing through the carbon filter. Airflow is measured with a calibrated orifice, and the pressure drop across the carbon filter is measured with a Magnahelix gage. A Miran infrared analyzer is used to measure the DMSO concentration before and after the carbon filter.

The procedure for measuring the carbon efficiency consists of generating a known concentration of DMSO vapor in an air stream, passing the airflow through the carbon filter, and measuring the DMSO vapor concentration before and after the canister. Figure 16 shows the carbon canister with a fresh charge of carbon being inserted into the canister housing. Figure 17 shows a photograph of an operator measuring the efficiency of the carbon canister. The pressure drop across the carbon canister was 0.49 inches water at 35 cfm. At 35 cfm, the downstream concentration was less than 1 ppm with an upstream concentration of 100 ppm, yielding an efficiency greater than 99%. The test was repeated after removing the carbon canister and dropping it four times at a height of 2 inches to simulate rough handling. A small amount (50 grams) of carbon spilled from the canister. (The carbon loss was due to the retaining screen pulling loose from the bottom plate when the canister was dropped. This design defect was eliminated after the test by welding the retaining screen to the bottom plate.). Efficiency measurements after the drop test were the same as before. A final efficiency test was conducted at 4 cfm and yielded the same concentration measurements and efficiency greater than 99%. Thus, we concluded our carbon filling procedure was satisfactory and yields efficiency greater than 99%.

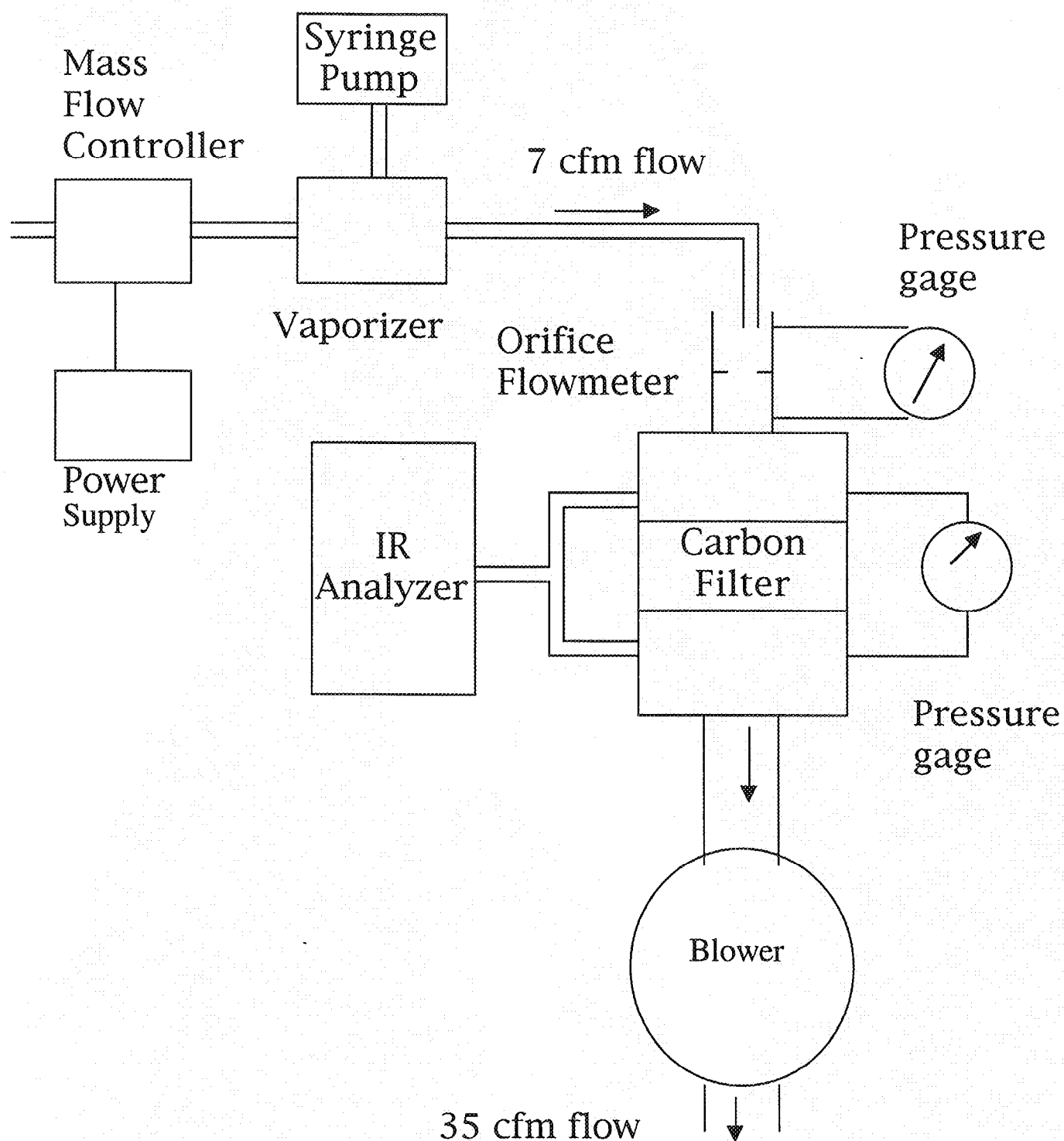


Figure 15. Schematic of the experimental apparatus for measuring the efficiency of the carbon canister in removing DMSO vapor.

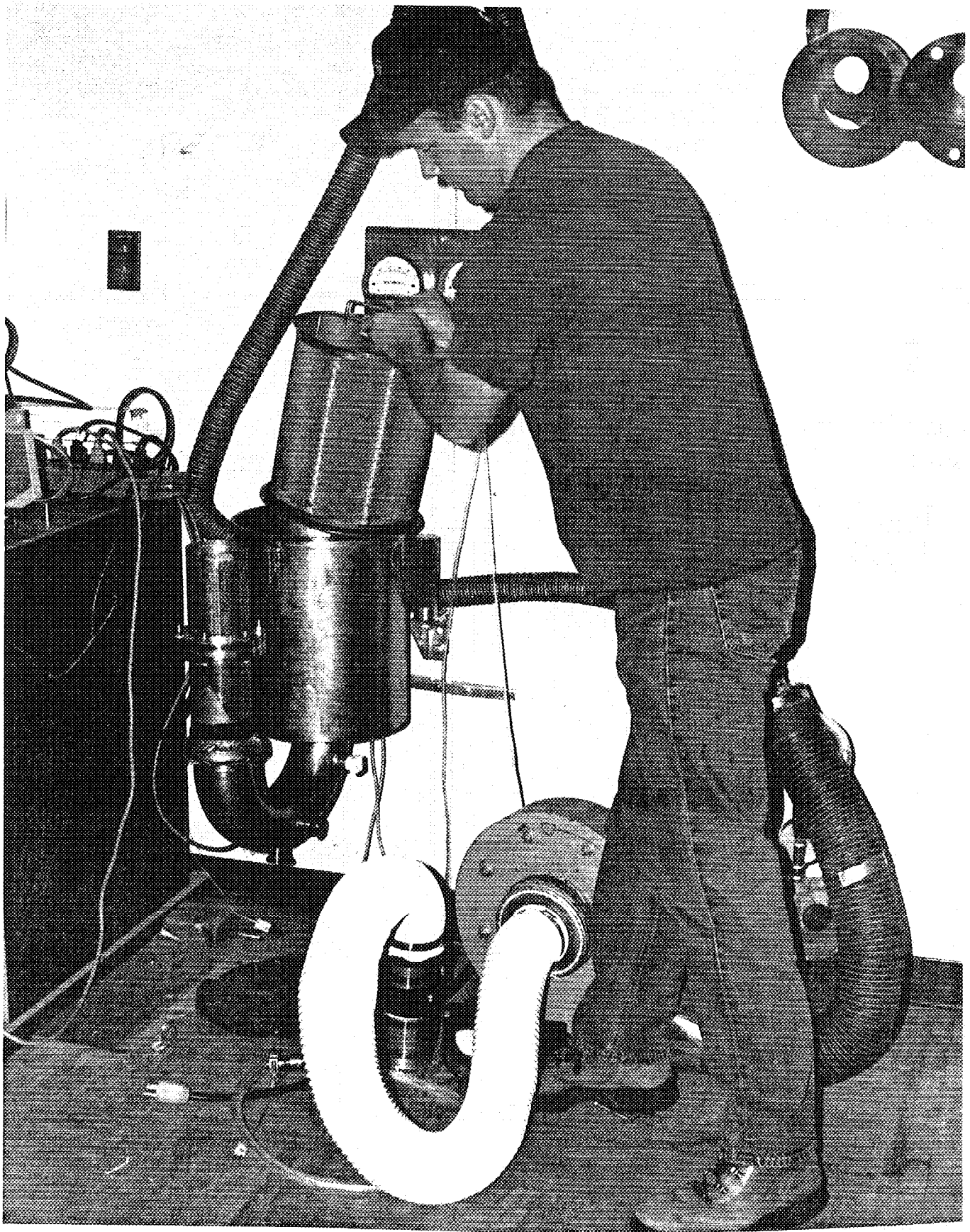


Figure 16. Photograph showing the carbon filter being inserted into the exhaust system housing.

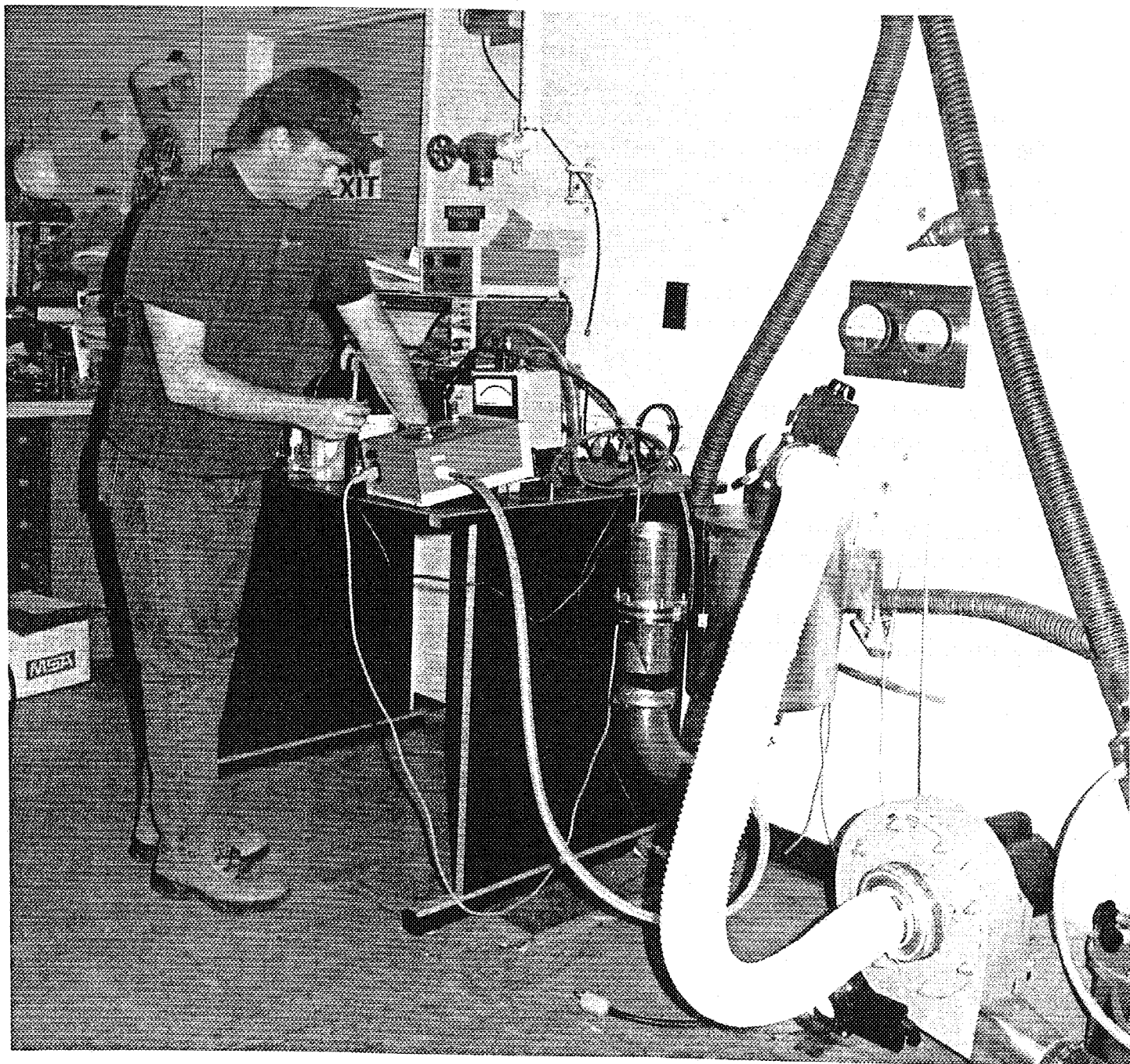


Figure 17 Photograph showing the experimental test apparatus for measuring the efficiency of the carbon filter for removing DMSO vapor.

Acknowledgment

We gratefully acknowledge the help of Mr. Jeff Oh for designing and fabricating the carbon filter canister and to Mr. Robert Ramos for help in testing the carbon canister.

References

1. Bergman, W, Wilson, K, Staggs, W, and Wapman, D, Lopez, R, and LeMay, J., "Degradation of HEPA filters exposed to DMSO" in Proceedings of the 24th DOE/NRC Nuclear Air Cleaning Conference, Available from NTIS, Springfield, VA, M. First, Ed., CONF-96 , pp., (1997).
2. Yoon, Y.H. and Nelson, J.H., "Application of gas adsorption kinetics: A theoretical model for respirator cartridge service life" Am. Ind. Hyg. Assoc. J. 45(8): 509-516 (1984).
3. ASME N509-1989, "Nuclear Power Plant Air-Cleaning Units and Components" American Society of Mechanical Engineers, 345 47th Street, New York, NY 10017, (1989).
4. ASME AG-1-1994, "Code on Nuclear Air and Gas Treatment", American Society of Mechanical Engineers, 345 47th Street, New York, NY 10017, (1994).
5. Martin, D., Hauthal, H.G., and Halberstadt, E.S., "Dimethyl Sulfoxide", Van Nostrand Reinhold Company Ltd., Berkshire, England (1975).